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(54) **OXYDE MIXTE DE SILICIUM ET D'ALUMINIUM**
(54) **SILICON-ALUMINIUM MIXED OXIDE**

(57) A silicon-aluminium mixed oxide produced by flame hydrolysis and having a composition of from 1 to 99.999 wt.% Al_2O_3 , the remainder being SiO_2 . The mixed oxide exhibits an amorphous structure in the X-ray diffraction pattern and consists of intergrown primary particles. In these primary particles crystallites are present; these crystallites having sizes of between one and 200 nanometres and the specific surface area of the powder being between 5 and 300 m^2/g . The silicon-aluminium mixed oxide is produced by a process wherein silicon halide and aluminium halide are vaporised in a particular ratio to one another, and are homogeneously mixed with air, oxygen and hydrogen in a mixing unit by means of a carrier gas. This mixture undergoes combustion in a burner of known construction and, after the separation of the solids from the vapour phase, any traces of halide possibly adhering to the product are separated off in a further processing step by means of moist air at elevated temperature.



Abstract

A silicon-aluminium mixed oxide produced by flame hydrolysis and having a composition of from 1 to 99.999 wt.% Al_2O_3 , the remainder being SiO_2 . The mixed oxide exhibits an amorphous structure in the X-ray diffraction pattern and consists of intergrown primary particles. In these primary particles crystallites are present; these crystallites having sizes of between one and 200 nanometres and the specific surface area of the powder being between 5 and 300 m^2/g . The silicon-aluminium mixed oxide is produced by a process wherein silicon halide and aluminium halide are vaporised in a particular ratio to one another, and are homogeneously mixed with air, oxygen and hydrogen in a mixing unit by means of a carrier gas. This mixture undergoes combustion in a burner of known construction and, after the separation of the solids from the vapour phase, any traces of halide possibly adhering to the product are separated off in a further processing step by means of moist air at elevated temperature.

Silicon-Aluminium Mixed Oxide

This invention relates to a silicon-aluminium mixed oxide, a process for the production thereof as well as the use as a polishing agent in dispersions which are used, for
5 example, for polishing electronic components, in particular for CMP applications.

The patent specification EP-0 585 544 discloses pulverulent silicon-aluminium mixed oxides produced by flame hydrolysis which are of amorphous structure and have a composition of
10 from 65 to 72.1 wt.% Al_2O_3 , and from 27.9 to 35 wt.% SiO_2 and a BET surface area of between 20 and 200 m^2/g .

The invention provides a silicon-aluminium mixed oxide powder produced by flame hydrolysis and having a composition of from 1 to 99.999 wt.% Al_2O_3 , preferably 40
15 to 80 wt.% Al_2O_3 , the remainder being SiO_2 , wherein the powder exhibits an amorphous structure in the X-ray diffraction pattern and consists of intergrown primary particles, and in these primary particles crystallites are present, and these crystallites have sizes of between one
20 and 200 nanometres and the specific surface of the powder is between 5 and 300 m^2/g , preferably between 50 and 150 m^2/g .

The invention also provides a process for the production of the silicon-aluminium oxide powder produced by flame
25 hydrolysis according to the invention, wherein silicon halide and aluminium halide are vaporised in a particular ratio to one another and are homogeneously mixed with air, oxygen and hydrogen in a mixing unit by means of a carrier gas, this mixture undergoes combustion in a burner of known
30 construction and, after the separation of the solids from the vapour phase, any traces of halide possibly adhering to the product are separated off in a further processing step by means of moist air at elevated temperature.

It has now been found that silicon-aluminium mixed oxides according to the invention which are made into a dispersion exhibit outstanding properties as polishing agents.

- 5 These dispersions can be used in particular for polishing in the electronics industry (CMP).

The silicon-aluminium mixed oxide according to the invention can also be used as a filler, as a supporting
10 material, as a catalytically-active substance, as ceramic raw material, in the electronics industry, as filler for polymers, as starting material for the production of glass or glass coatings or glass fibres, as auxiliary separating agents, in the cosmetics industry, as absorbent material,
15 as an additive in the silicone and rubber industry, for adjusting the rheology of liquid systems, for heat-protective stabilization, as heat-insulating material, as flow-control agents, in the dental industry, as auxiliary agents in the pharmaceutical industry, in the paint
20 industry, in PET-film applications, in fluorescent tubes, as a starting material for the production of filter ceramics or filters, in powders for toners, as rust inhibitors, as agents for the film-coating of polyethylene (PET) and polyvinyl acetate, in inks and in battery
25 separators.

Characteristics of the oxide powders according to this invention are illustrated in the accompanying drawings, in which:

30

Figure 1 shows X-ray diffraction patterns of these powders; and

Figure 2 is a high-resolution electron micrograph of
35 primary particles of the powders.

Examples

Utilizing the known burner arrangement described in Example
5 1 of EP 585,544, the following quantities are used for the
production of the mixed oxide.

Example 1:

10 1.6 Nm³/h hydrogen from the burner or from the reaction
together with 5 Nm³/h air and 1.70 kg/h of previously
vaporised SiCl₄, are mixed together. Into this mixture,
which is at a temperature of about 200°C, is fed an
additional 2.5 kg/h of gaseous AlCl₃ (which has previously
15 been vaporised at about 300°C). This mixture undergoes
combustion in a flame tube, while an additional 12 Nm³/h of
air is fed into this flame tube.

After the material has passed through the flame tube, the
20 resulting powder is separated, in a filter or cyclone, from
the gases containing hydrochloric acid, and adhering traces
of hydrochloric acid are separated from the resulting mixed
oxide by treatment at elevated temperature.

25 The analytical data for the mixed oxide is as follows:

Specific BET surface area 56 m²/g, pH of a 4 per cent
dispersion 4.52, bulk density 49 g/l, tamped density 59
g/l. Composition of the powder: 63.6 wt.% Al₂O₃, 36.3
30 wt.% SiO₂.

Example 2:

1.2 Nm³/h hydrogen from the burner or from the reaction
35 together with 6 Nm³/h air and 1.70 kg/h of previously

vaporised SiCl_4 are mixed together. Into this mixture, which is at a temperature of about 200°C , is fed an additional 2.5 kg/h of gaseous AlCl_3 (which has previously been vaporised at about 300°C). This mixture undergoes
5 combustion in a flame tube, while an additional $12 \text{ Nm}^3/\text{h}$ of air is fed into this flame tube.

After the material has passed through the flame tube, the resulting powder is separated, in a filter or cyclone, from
10 the gases containing hydrochloric acid, and adhering traces of hydrochloric acid are separated from the resulting mixed oxide by treatment at elevated temperature.

The analytical data for the mixed oxide is as follows:

15

Specific BET surface area $99 \text{ m}^2/\text{g}$, pH of a 4 per cent dispersion 3.9, bulk density 39 g/l, tamped density 48 g/l. Composition of the powder: 64.3 wt.% Al_2O_3 , 35.52 wt.% SiO_2 .

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The powders are additionally characterized by having the following features:

X-ray diffraction patterns of these powders (Figure 1 of
25 Example 1) show that there are virtually no crystalline phases in the powder.

At the same time a high-resolution electron micrograph (Figure 2) shows that the primary particles of the powders
30 contain partially crystalline regions, which probably are responsible for the good polishing performance of dispersions prepared from these mixed oxides.

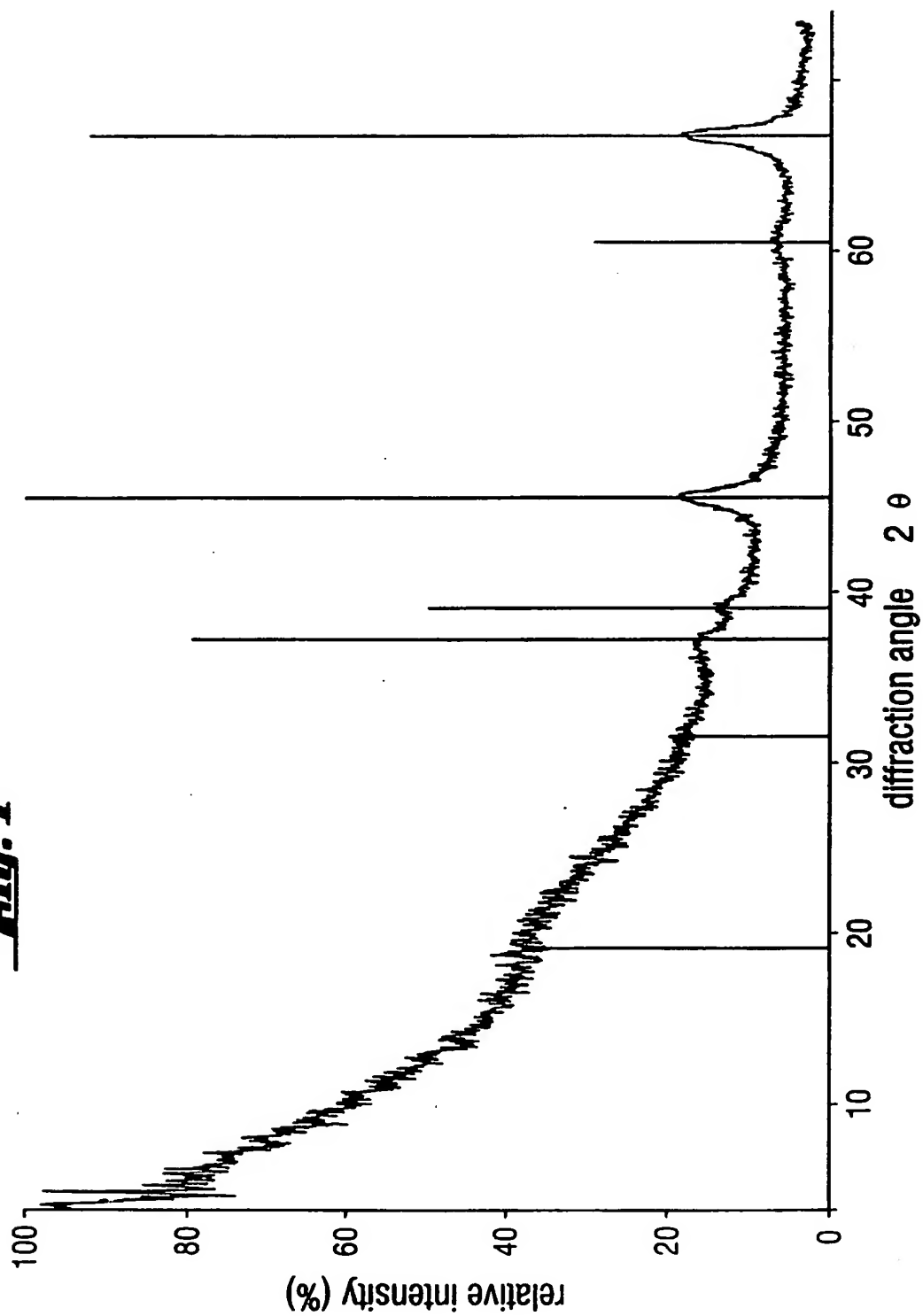
The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A silicon-aluminium mixed oxide powder produced by flame hydrolysis and having a composition of from 1 to 99.999 wt.% Al_2O_3 , the remainder being SiO_2 ; in which the powder exhibits an amorphous structure in an X-ray diffraction pattern, and the powder consists of intergrown primary particles and crystallites are present in the primary particles; and wherein the crystallites are between one and 200 nanometres in size, and the specific surface area of the powder is between 5 and 300 m^2/g .

2. A process for the production of the silicon-aluminium mixed oxide produced by flame hydrolysis as claimed in claim 1, comprising the steps of:
vaporizing silicon halide and aluminium halide in a particular ratio to one another;
homogeneously mixing the silicon halide and the aluminium halide with air, oxygen and hydrogen in a mixing unit by means of a carrier gas;
combusting the mixture in a burner;
separating solids from vapour; and
separating off any traces of halide by means of moist air at elevated temperature.

3. The use of the silicon-aluminium mixed oxide produced by flame hydrolysis as claimed in claim 1 for the production of dispersions which are used for polishing.

4. The use of the silicon-aluminium mixed oxide produced by flame hydrolysis as claimed in claim 1 as a filler, as a supporting material, as a catalytically-active substance, as ceramic raw material, in the electronics industry, as a filler for polymers, as a starting material for the production of glass or glass coatings or glass fibres, as auxiliary separating agents, in the cosmetics industry, as an absorbent material, as an additive in the silicone and rubber industry, for adjusting the rheology of liquid systems, for heat-protective stabilization, as heat-insulating material, as flow-control agents, in the dental industry, as auxiliary agents in the pharmaceutical industry, in the paint industry, in PET-film applications, in fluorescent tubes, as starting material for the production of filter ceramics or filters, in powders for toners, as rust inhibitors, as agents for film-coating of polyethylene (PET) and polyvinyl acetate, in inks, and in battery separators.

Fig. 1

Markus + Clerk.



10nm

Fig. 2

Markus-Chek